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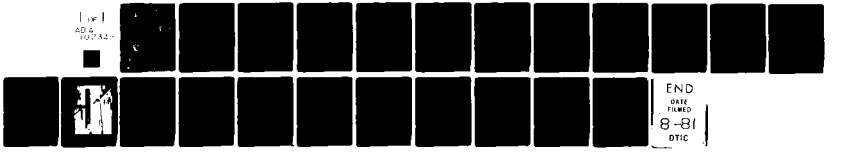

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SOME EXPERIENCES WITH THE USE OF CASTOR OIL AS A DIELECTRIC

Vernon A. Nieberlein
Directed Energy Directorate
US Army Missile Laboratory

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Experiences with the use of large volumes of castor oil for dielectric purposes are presented. These experiences include corrosion of brass parts by the oil, drying of the oil, and the determination of water in the two-phase system of castor oil in contact with mineral oil. Detailed design of a packed column for the purpose of drying castor oil is also presented.

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NOMENCLATURE

A	area, ft^2
C_L	water content of the main body of oil, moles ft^{-3}
C_{Lo}	initial water content of the oil, moles ft^{-3}
C^*	equilibrium concentration of water in the oil, moles ft^{-3}
g	gravitational constant, cm sec^{-2}
G_L	mass flow rate of the liquid phase, lb ft^{-2} hr^{-1}
H	Henry's constant, lb_f ft^{-2} (moles $ft^{-3})^{-1}$
H_G	height of a transfer unit referred to interfacial gas film resistance, ft
H_L	height of a transfer unit referred to interfacial liquid film resistance, ft
H_{OG}	height of a transfer unit referred to overall gas phase resistance, ft
K	overall mass transfer coefficient, ft^3 hr^{-1}
k_G	mass transfer coefficient for water in gas, moles ft^{-2} hr^{-1} $(lb_f$ $ft^{-2})^{-1}$
k_L	mass transfer coefficient for water in oil, moles ft^{-2} hr^{-1} (moles $ft^{-3})^{-1}$
L	molar flow rate of the liquid phase, moles ft^{-2} hr^{-1}
L'	molar flow rate of totally dry oil, moles ft^{-2} hr^{-1}
m	slope of the equilibrium line
N_{OG}	number of transfer units referred to overall gas phase resistance
N_{Sc}	Schmidt number for oil
n	exponent in Equation 16
p_G	partial pressure of water in the main body of gas, lb_f ft^{-2}
p_i	partial pressure of water at the interface, lb_f ft^{-2}
p_o	atmospheric pressure, dynes cm^{-2}
Q	volume, ft^3
R	bubble radius, cm

t	time, hr
V	molar flow rate of gas, moles ft^{-2} hr^{-1}
V'	molar flow rate of totally dry gas, moles ft^{-2} hr^{-1}
v_t	terminal velocity of a bubble, cm sec^{-1}
W_G	transport rate of water through nitrogen, moles hr^{-1}
W_L	transport rate of water through oil, moles hr^{-1}
x	mole fraction of water in oil
y	mole fraction of water in nitrogen
y^*	equilibrium mole fraction of water in nitrogen
z	vertical distance, cm
β	transfer factor in Equation 16, ft^{n+1}
μ	viscosity, g cm^{-1} sec^{-1}
μ_L	viscosity of oil in Equation 16, lb ft^{-1} hr^{-1}
ρ_L	density of the liquid surrounding a bubble, g cm^{-3}
ρ_s	density of a spherical bubble, g cm^{-3}

ACKNOWLEDGEMENT

Grateful acknowledgement is made to Jimmy D. Burnett of the Propulsion Directorate, Army Missile Laboratory, for chemical and infrared analysis of castor oil.

I. INTRODUCTION

Castor oil has been chosen as a dielectric liquid for the ERAM project. It has also been used in various capacitors in other projects in the Directed Energy Directorate. Some of the experiences with this fluid merit recording.

The original choice of this oil was based on several worthwhile features. It is a vegetable oil, and therefore is not an environmental hazard. There is also permitted the advantage of working with hands and arms submerged, if necessary, as the oil appears to soften and condition the skin, rather than irritate. Overriding are its electrical properties[1] in high-frequency or pulsed applications, and the cost is relatively reasonable. A possible disadvantage is its high viscosity which makes pumping difficult, especially on cold days.

II. CORROSION

Some parts of the ERAM capacitor system were made from 70-30 cartridge brass. After prolonged contact with castor oil, the brass corroded slightly to give a green coloration to the oil coating. An attempt was made to duplicate these conditions and also provide electrolytic couples to accelerate corrosion. For this purpose, a specimen was made as shown in Figure 1. The two brass pieces in this specimen were immersed in castor oil, then screwed together to provide an interface where air would be excluded. The specimen also provided surfaces which would be in contact with air.

Approximately nine weeks were required for the brass to show initial signs of corrosion. Where the specimen was partially submerged in castor oil, the brass was definitely darker than above the liquid level. Also, one drop of oil on the brass above the liquid level had turned green. There was no evidence of electrolytic corrosion as there was no halo of any type around the cap screw nor around the zinc-plated nut.

III. MAINTAINING DRY CASTOR OIL

The effect of moisture on the dielectric strength of organic liquids is well known[2]. The dielectric strength of mineral oil, for example, will increase from 20 kV cm^{-1} to 73 kV cm^{-1} if the water content is reduced from 75 ppm to 15 ppm and the electrical stress is provided by 60 Hz ac. While figures for castor oil are not readily available, it is known to follow a similar pattern[3]. Thus, it becomes important to maintain the water content of the castor oil at the lowest possible level. Since this oil is extremely hygroscopic, this is a difficult task.

As purchased from the vendor, castor oil is partially dehydrated. This is different from partially dried. Drying, by definition, is the removal of dissolved or interstitial water from the aggregate of the various molecules which make up castor oil. Dehydration, on the other hand, is the removal of hydrogen and hydroxyl groups, in the ratio of one-to-one, from the molecules themselves, thus creating a different chemical structure. Figure 2 shows the structure of glyceryl triricinoleate, the principal component of castor oil. The illustration also shows the manner in which dehydration takes place. In the dehydration process, the oil is heated to the neighborhood of 300°C under an absolute pressure of 1-2 torr in the presence of a catalyst. This treatment causes the hydroxyl groups of the glyceryl triricinoleate to react with the neighboring hydrogen on either side, thus forming water (which is removed) and at the same time creating a double bond in either the 11 or 12 position in the chain. Care is taken not to overdehydrate which would form a drying oil, that is, one which would harden to a solid when exposed to air. With the removal of water from the molecule, the potential exists for the uptake of free water to again form glyceryl triricinoleate. To what extent this actually happens is not known, but it is known that partially dehydrated castor oil direct from the vendor still contains over 300 parts per million of free water and on exposure to humid air this value may rise to several thousand ppm. To remove free water, several drying systems were used for the oil in the ERAM equipment.

The first system was a simple bubbler type, which consisted of an aluminum tube laid on the bottom of the oil vat. At measured intervals there was a hole in the side of the tube covered and sealed with Tygon tubing. The Tygon had a small slit through the wall to allow gas to pass out and also to inhibit oil from passing in. The aluminum tubing was pressurized with nitrogen and the bubbles floated upward through approximately two feet of oil.

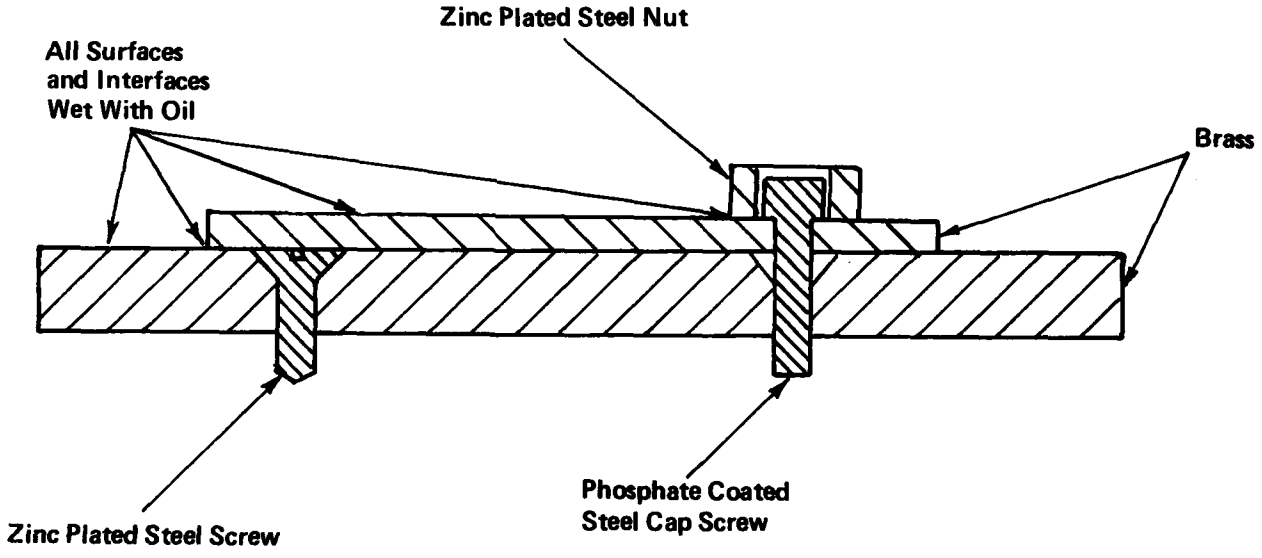

Figure 1. Brass corrosion specimen.

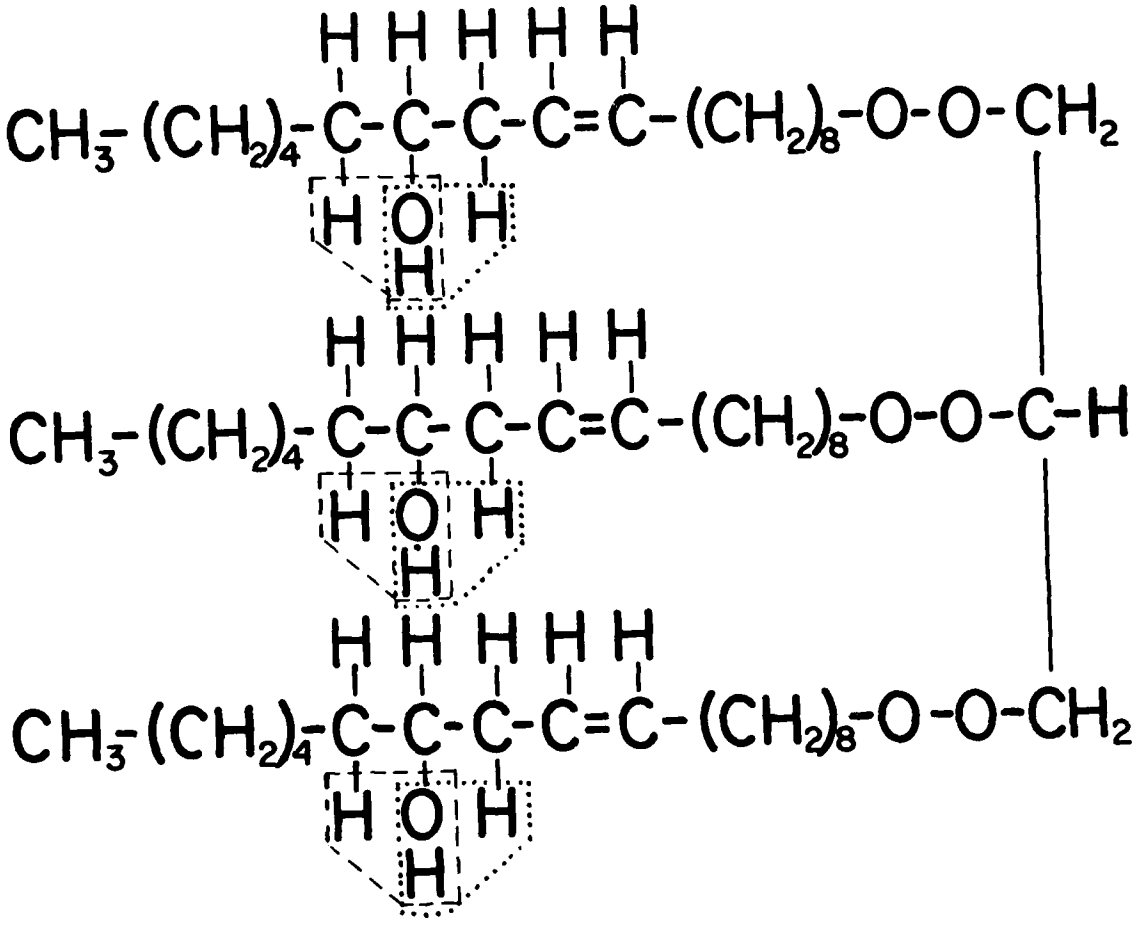

Figure 2. Dehydration of glyceryl triricinoleate.

A brief analysis of this system was made, using the model shown in Figure 3. This is a two-film model, that is, it depicts all of the bubbles in the tank at any instant to be coalesced to form a single gas volume to the right of the liquid-gas interface. Two films are adjacent to the interface – one in each phase. In the main body of oil, concentration of water is C_L. In the oil film next to the interface, this concentration drops off to some equilibrium value C_i. On the gas side, the partial pressure of water vapor in equilibrium with C_i is p_i. This partial pressure falls off to that of the main body of gas p_G. The moles per unit time transferred from the main body of liquid through the liquid-side film to the interface is

$$W_L = Ak_L(C_L-C_i) = \frac{(C_L-C_i)}{\frac{1}{Ak_L}} = \frac{\text{driving force}}{\text{resistance}}, \quad (1)$$

and the moles per unit time transferred from the interface through the gas-side film to the main body of gas is

$$W_G = Ak_G(p_i-p_G) = \frac{(p_i-p_G)}{\frac{1}{Ak_G}} = \frac{\text{driving force}}{\text{resistance}}, \quad (2)$$

where A is the total interfacial area, and k_L and k_G are the mass transfer coefficients for the liquid and gas, respectively. From Henry's Law,

$$p_i = HC_i \quad , \quad (3)$$

and

$$p_G = HC^* \quad , \quad (4)$$

where H is Henry's constant and C^* is a hypothetical concentration of water in the liquid that would be in equilibrium with p_G if p_G extended all the way to the interface. Substituting Equations 3 and 4 into Equation 2,

$$W_G = \frac{(C_i-C^*)}{\frac{1}{HAk_G}} \quad . \quad (5)$$

The overall transfer rate then is the total driving force divided by the total resistance,

$$W_t = \frac{C_L-C^*}{\frac{1}{Ak_L} + \frac{1}{HAk_G}} = K(C_L-C^*) \quad . \quad (6)$$

At 77° K, the temperature of liquid nitrogen, the vapor pressure of ice is 2.6×10^{-11} atm or if the boil-off gas from liquid nitrogen is used as the purge gas $C^* \doteq 0$. This value of C^* does not substantially deviate from 0 in the gas bubble because of the relatively short float times of medium-size bubbles. The following calculation confirms that the float time is relatively short for typical bubbles.

The various forces acting on a solid sphere which is sinking or rising through a liquid is given[4] as

$$\frac{4}{3}\pi R^3 \rho_S g = \frac{4}{3}\pi R^3 \rho_L g + 2\pi\mu R v_t + 4\pi\mu R v_t \quad , \quad (7)$$

$$\text{(weight)} = \text{(buoyancy)} + \text{(form drag)} + \text{(friction drag)}$$

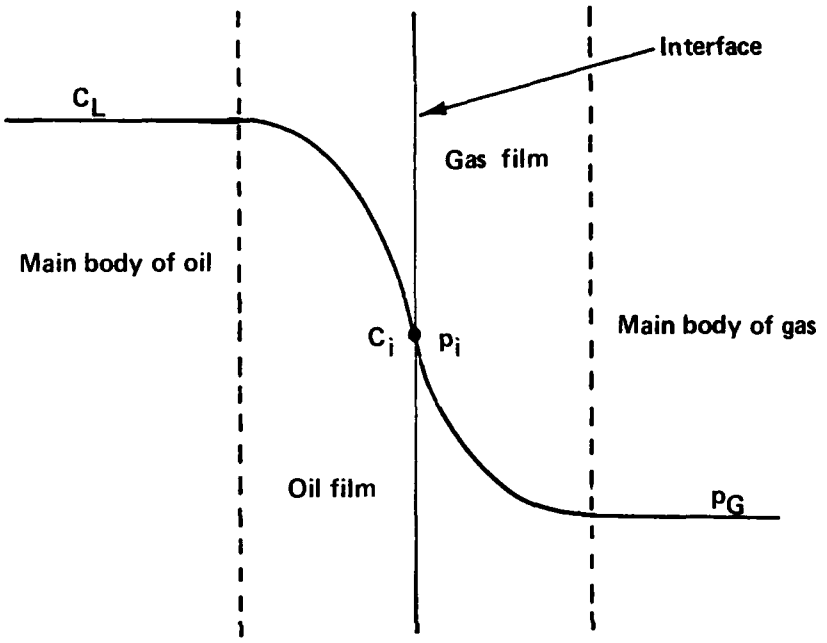

Figure 3. Two-film model of drying by the use of bubbles.

where R is the radius of the sphere, ρ_L is the density of the liquid, μ is the viscosity of the liquid, v_t is the terminal velocity of the sphere, and g is the gravitational constant. In the case of a bubble, ρ_S, the density of the sphere, is relatively insignificant compared to ρ_L, causing the first term in Equation 7 to vanish. Also, in the case of a bubble, near the gas-liquid interface, virtually all of the shear movement will be in the gas phase because its viscosity is five orders of magnitude less than castor oil. Thus, the μ in the last term becomes the viscosity of the gas in the bubble, and this fact makes the fourth term insignificant compared to the third term, causing the fourth term to also vanish.

The value for R is not quite constant, but increases as the bubble rises because of a decrease in hydrostatic pressure. The amount of this increase in R is given by the relationship,

$$R^3 = R_o^3 \frac{\rho_L z g + p_o}{p_o}, \tag{8}$$

where z is the vertical distance above the bubble starting point, p_o is the ambient atmospheric pressure, and R_o is the initial radius of the bubble. Substituting Equation 8 into Equation 7 and solving for terminal velocity,

$$v_t = \frac{2\rho_L g}{3\mu} R_o^2 \left[\frac{\rho_L z g + p_o}{p_o}\right]^{2/3}. \tag{9}$$

Using values of v_t from Equation 9, the times required for various size bubbles to float upward through 60 cm of oil are shown in Table I.

TABLE I

Bubble Diameter	Bubble Lifetime
1 cm	3.7 sec
1 mm	6.2 min
0.1 mm	10.4 hrs

Because the bubble lifetimes are of the order of a few seconds, it may be presumed that the value of C* never appreciably deviates from 0 in Equation 6. Since W_t is the overall total transfer rate of water out of the oil, a balance on the water content of the oil tank can be made which leads to

$$KC_L = -Q \frac{dC_L}{dt}, \tag{10}$$

where Q is the volume of the oil tank. The solution of Equation 10 is

$$C_L = C_{Lo} e^{\frac{Kt}{Q}}, \tag{11}$$

where C_{Lo} is the initial concentration of water in the oil. A chemical analysis to determine C_{Lo} and another analysis after t hours to determine C_L could provide the value for K Q. However, this was not done because (a) there was not an adequate cover on the oil vat, which allowed humidity from the air to be absorbed by the castor oil while the nitrogen bubbler system was removing the water and (b) the area of the oil in contact with humid air was orders of magnitude greater than the area of the oil in contact with dry nitrogen in all of the bubbles. A partial

solution of the problem was attempted by covering the oil vat with 6-mil polyethylene sheet material, but not in a gas-tight manner. Later a rigid cover was built of 1/4-inch Lexan, more closely approaching gas-tightness. In the meantime an attempt was made to make the oil-nitrogen contact area more competitive to the oil-humid air contact area. To do this a drying column was designed and built.

IV. DRYING COLUMN DESIGN AND CONSTRUCTION

A column for the drying of castor oil was designed around available materials and space, rather than around desired performance. Polyvinyl chloride pipe, 12 inches in diameter, was available for the body and polyvinyl chloride slabs were available for the ends and internal antichanneling rings. Screens and Tellerettes were available for internal packing. Available space to the top of the room was 14 feet. Since the effluent from the drying column had to run by gravity to the opposite side of the room for reentry into the oil vat, the column was put on a stand seven feet high, leaving seven feet of space for the installation of the dryer. The dryer stand was also a section of the same polyvinyl chloride pipe, 12 inches in diameter and seven feet long. This equipment is shown in Figure 4. The actual overall length of the dryer was six feet, allowing some space for a pipe to bring oil into the top. Of the six feet overall length, the packed volume was five feet long with a six-inch section at the top and bottom for distribution and collection, respectively, of the oil as it entered and left the column. A drawing of the equipment is shown in Figure 5. A Tellerette is shown in Figure 6.

Data show[2] that dielectric oils, once wet, will no longer dry to zero percent moisture even if the gas in contact with them has zero percent relative humidity. Based on this and a realistic gas flow upward, an example design calculation is made with the following assumptions:

(a) N_2 flows upward at 20 ft^3hr^{-1}, entering at the bottom of the column at zero percent relative humidity.

(b) Oil flows downward at the rate of one drum (55 gallon) per hour, entering at the top of the column and having a water content of 4000 ppm by weight.

(c) Oil leaves the bottom of the column reduced in water content by 40 ppm.

(d) Castor oil has a molecular weight of 300.

Using L and V as the molar flow rates of liquid and gas, respectively, and x and y as the mole fraction of water in the liquid and gas, respectively, simple material balances are made and the results are shown in Figure 7. Subscripts 1 and 2 refer to the conditions at the bottom and top of the column.

The number of transfer units, a measure of the difficulty of separation, is now calculated. For dilute solutions such as this problem presents, simplifications in the rigorous analytical equations can be made which lead to

$$N_{OG} = \int_{y_1}^{y_2} \frac{dy}{y^*-y} , \tag{12}$$

where N_{OG} is the number of transfer units derived from the overall gas phase resistance and y^* is a hypothetical mole fraction of water in the gas phase which would be in equilibrium with the real water content of the liquid phase. To obtain y^* an equilibrium line must be drawn to give the relationship of the mole fraction of water in the oil (x) which is in equilibrium with any mole fraction of water in the nitrogen (y). Several chemical analyses for water in that castor oil which came fresh from the gas-tight shipping drum and that which had prolonged exposure to humid air led to the following problem conditions:

(a) Castor oil is in equilibrium with saturated nitrogen when the oil contains 5300 ppm of water.

(b) Castor oil is in equilibrium with bone dry nitrogen when the oil contains 300 ppm of water.

(c) The relationship is a straight line.

Besides an equilibrium line (x vs y), an operating line (also x vs y) must be drawn. This line gives the designed operating conditions, which must have the water content in the nitrogen less than the equilibrium value for water in the oil at the same point if transfer of water from the oil to nitrogen is to take place. To get the operating line, material balances from Figure 7 lead to the following equation for the operating line,

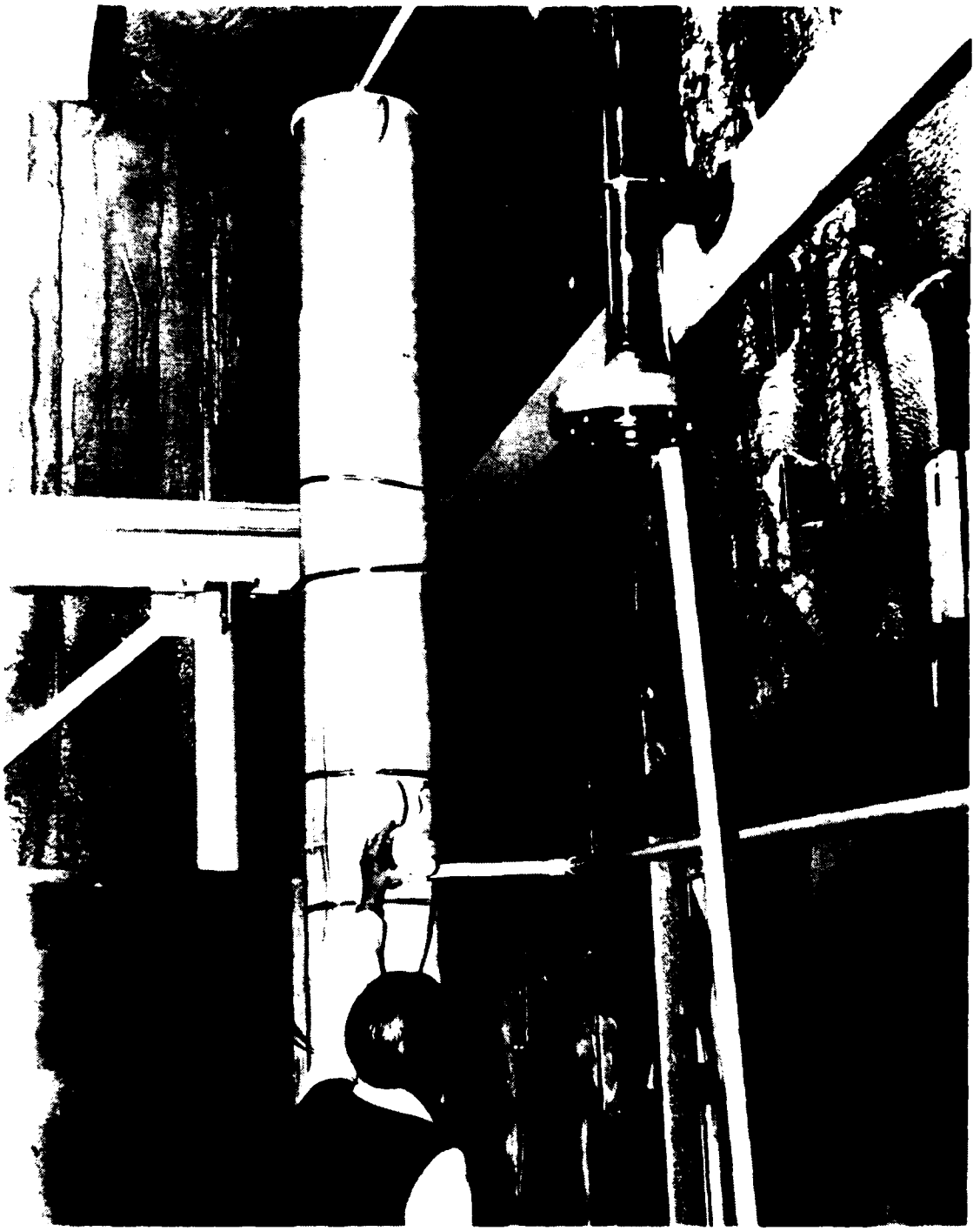

Figure 4. Drying column installation.

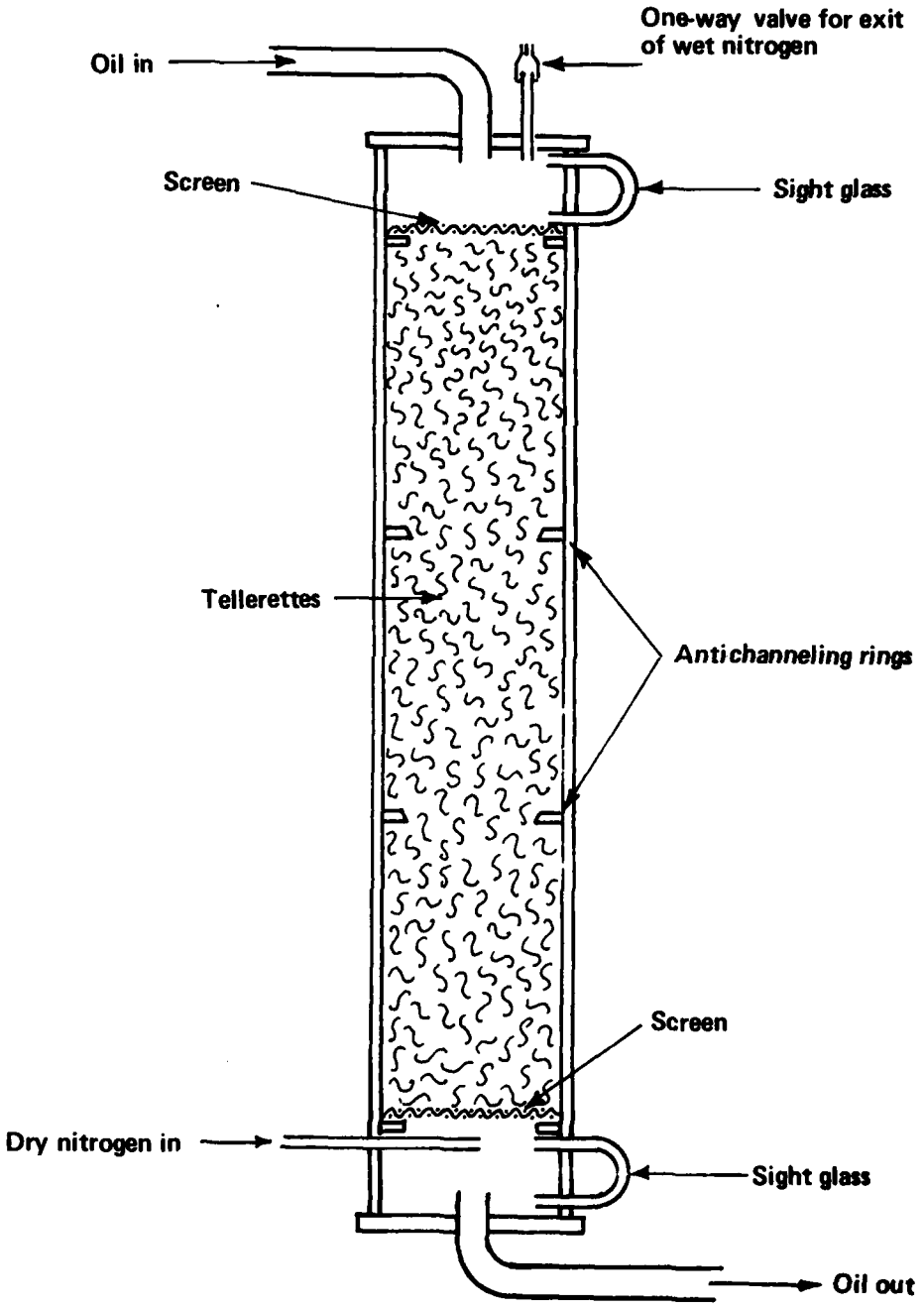

Figure 5. Details of the drying column.

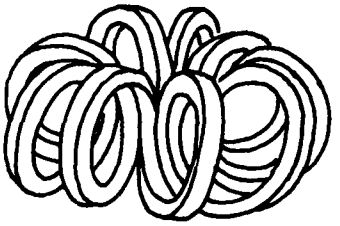

Figure 6. A Tellerette

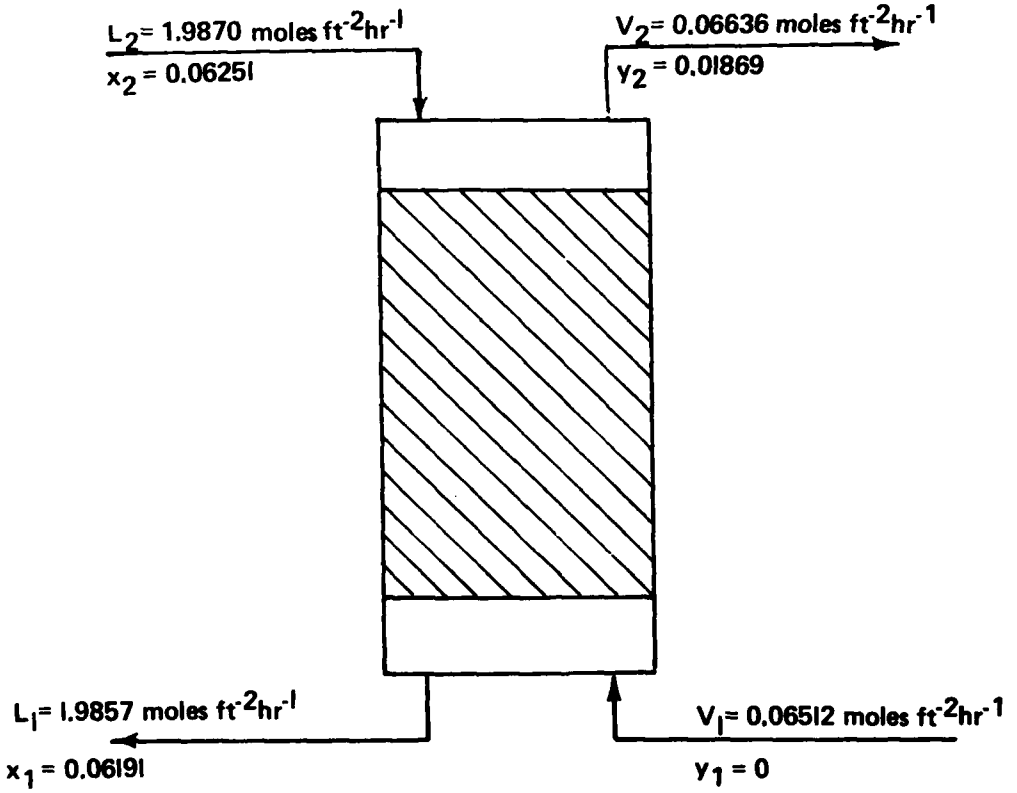

Figure 7. Material balance on the drying column.

$$V' \left(\frac{y_1}{1-y_1}\right) + L' \left(\frac{x}{1-x}\right) = V' \left(\frac{y}{1-y}\right) + L' \left(\frac{x_1}{1-x_1}\right) . \quad (13)$$

The V' and L' are values of V and L after the water content has been subtracted. Both the equilibrium line and the operating line are in Figure 8. For each x value in Figure 8 we designate the y value on the equilibrium line as y* and leave the y designation on the operating line as is, and thereby form a set of values for y*-y. A plot of y vs $1/y^*-y$ is given in Figure 9. Graphical integration is used to obtain the area under this curve, which gives the value for N_{OG} in Equation 12. Since the equilibrium curve is a straight line and the operating curve is extremely close to a straight line, a simplified equation might have been used instead of graphical integration,

$$N_{OG} = \frac{y_2-y_1}{(y^*-y)_{\log \text{ mean}}} . \quad (14)$$

For this example, Equation 12 was used and then checked by Equation 14.

While Equation 12 gives the number of transfer units necessary for the drying column design, the height of a transfer unit is still needed. This is designated as H_{OG}, where the subscript refers to the overall resistance in the gas phase. In the present problem the controlling resistance is in the liquid phase since water diffusion through castor oil is much slower than through nitrogen. These values are related as follows,

$$H_{OG} = H_G + \frac{mV}{L} H_L , \quad (15)$$

where m is the slope of the equilibrium line and H_G and H_L are the transfer unit heights referred to the interfacial resistance on the gas side and liquid side of the interface, respectively. $H_G \doteq 0$ since the liquid side resistance is controlling. H_L has been experimentally correlated by Sherwood and Holloway[5] as follows,

$$H_L = \beta \left[\frac{G_L}{\mu_L}\right]^n (N_{Sc})^{0.5} , \quad (16)$$

where β is a factor dependent on the type and size of column packing, G_L is the mass velocity of the liquid, μ_L is the liquid viscosity, and N_{Sc} is the Schmidt number for castor oil. Using the method of Wilke and Chang[6] for estimating the diffusion coefficient of water in castor oil, N_{Sc} was calculated to be 5.76×10^7, G_L was 558.8 lbs hr^{-1} ft^{-2}, and μ_L was 1730 lbs hr^{-1} ft^{-1}. The exponent n was estimated to be 0.22 (a value typical for Raschig ring packing) and β was estimated to be 0.01 (also typical for Raschig rings). Values for Tellerettes were not available, but should be somewhat similar to those for Raschig rings. Equation 16 gave a height for H_L of 59.19 ft and Equation 15 gave a height for H_{OG} of 0.70 ft. The product of H_{OG} and N_{OG} (from Equation 12) gave a column height of 2.0 ft. This means that for the design requirements given, the column functions only in the bottom two feet of height. Above this the nitrogen carries almost an equilibrium value of moisture (about 70% relative humidity) and thus a negligible transfer of water takes place from the oil to the nitrogen. Since the equilibrium line is almost a straight line, the slope m of Equation 15 is a constant. H_L is a function of L but not of V so that for a fixed oil flow, H_{OG} becomes directly proportional to V and, thus, the functioning part of the column height becomes directly proportional to V. The required nitrogen flow necessary to utilize the full five feet of column height available would be 50 ft^3 hr^{-1}. Flows of this magnitude are not available from a liquid nitrogen tank. The initial flow from such a tank is 20 ft^3 hr^{-1} but the boiling nitrogen soon cools the surroundings so that the gas flow levels off in the vicinity of 1.0 ft^3 hr^{-1}. Under this latter gas flow, the effluent from the dryer can be expected to have a water content only 2 ppm lower than the oil entering the dryer. A possible solution is to have a liquid nitrogen freeze trap installed for gaseous nitrogen being fed from a compressed gas cylinder into the dryer at 50 ft^3 hr^{-1}.

Water determinations were made on the castor oil using the Karl Fischer method. The first analysis was made after the oil vat had been in use for several weeks. The vat was covered with a polyethylene sheet but there was no attempt to make the cover gas-tight so the oil had picked up water to the extent of 1100 ppm. After 38 days

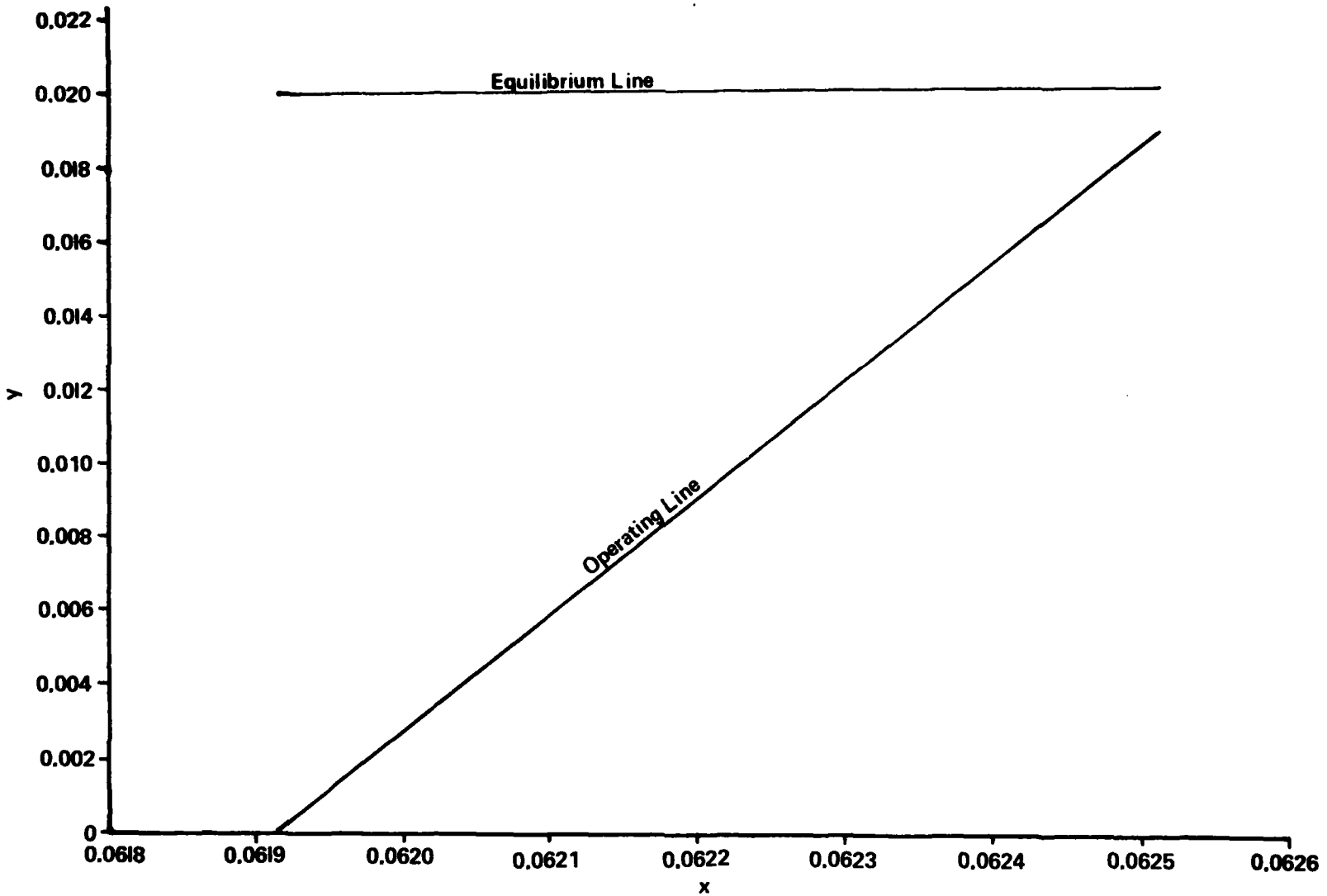

Figure 8. Equilibrium and operating lines.

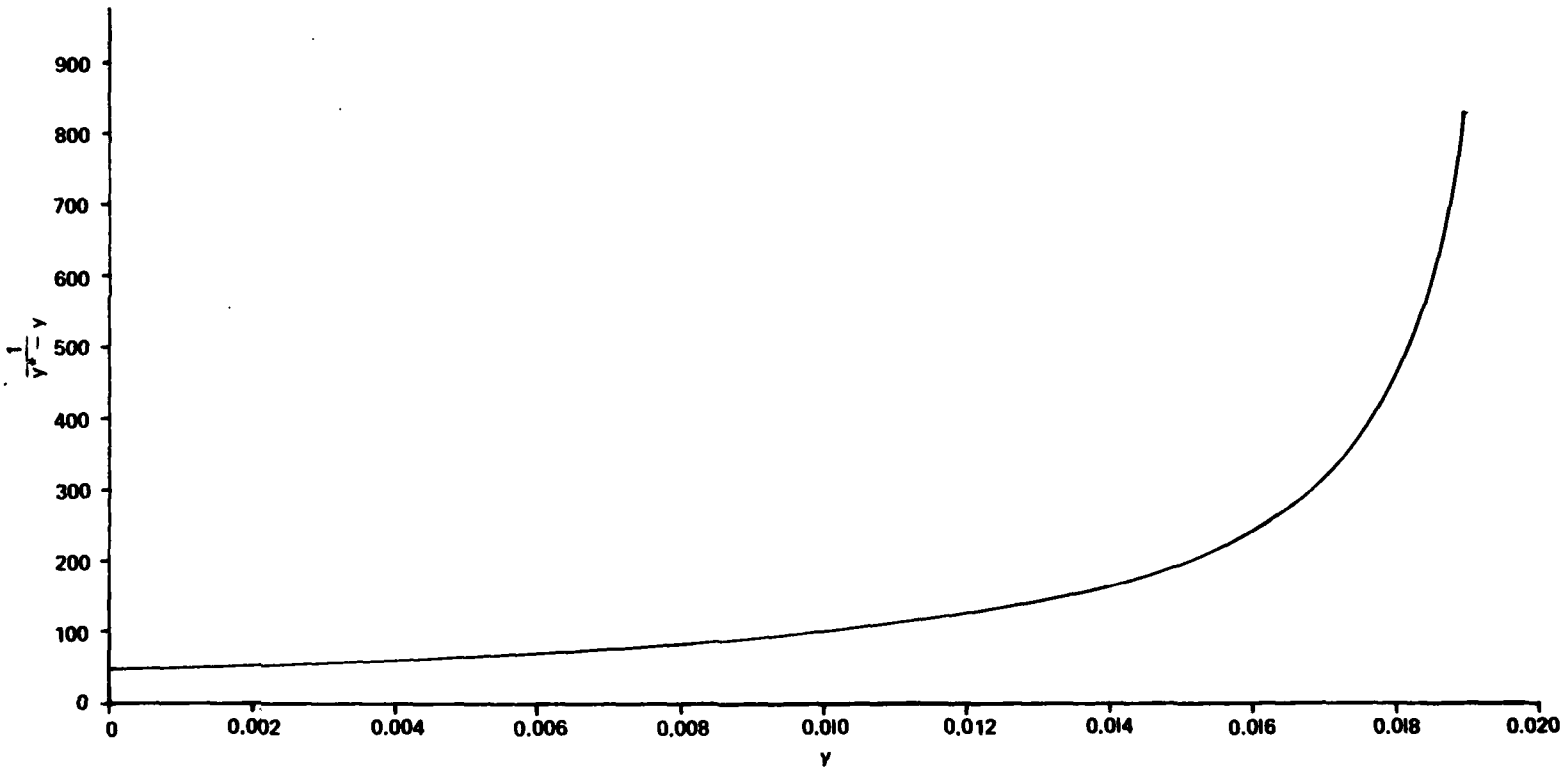

Figure 9. Mole fraction of water in nitrogen vs the reciprocal of the driving force.

the dryer was installed. On this day the oil in the vat analyzed 4200 ppm of water while the dryer effluent was 4100 ppm. The difference in water content was enough to show that the dryer was functioning, but not necessarily to the extent of removing 100 ppm of water. This is because the standard deviations in the vat analyses and the dryer analyses were 400 and 300 ppm, respectively. Figure 10 shows the history of the water content of the oil from the date of the dryer installation to 16 days later – the trend was definitely downward. After this, new oil was added. Modifications were subsequently made in the ERAM equipment which made it impractical, at least temporarily, to operate the dryer.

V. CASTOR OIL IN CONTACT WITH TRANSFORMER OIL

In the course of the work on water in the castor oil from the ERAM project, analyses were also made on dielectric oils from another project in the Directed Energy Directorate. The latter was a laser project and the equipment involved capacitors and transformers. In one case the sample of transformer oil contained globules of a second immiscible and heavier liquid. The infrared spectrometer showed the globules to be castor oil, probably from a leaky capacitor in the system. Analysis of the castor oil determined the water content to be 4600 ppm while the water in the transformer oil was only 190 ppm. This provided speculation that castor oil might be useful in a liquid-liquid extraction process to remove water from transformer oil.

VI. CONCLUSIONS

1. Castor oil slightly corrodes brass after nine weeks of exposure. Corrosion rates were not measured.
2. Castor oil must be sealed from humid air if dryness is to be maintained.
3. Some of the dissolved water can be removed from "wet" castor oil by contacting it with extremely dry nitrogen.
4. Because of the great differences in interfacial areas, a drying column is more effective than bubbling N_2 with one cm diameter bubbles.
5. In a drying column one foot in diameter, an oil flow of one drum per hour, and a nitrogen flow of 20 cubic feet per hour, the moisture content of the oil can be theoretically reduced from 4000 ppm to 3960 ppm. Chemical analyses were not conclusive enough to prove or disprove these figures, although a reduction in water content was demonstrated.
6. In the drying column described above, only the lower two feet of the column is effective, because of insufficient gas flow.
7. Castor oil with 4600 ppm of water maintains equilibrium with a separate liquid phase of transformer oil containing 190 ppm of water. This fact could lead to a process for extracting water from transformer oil with dry castor oil.

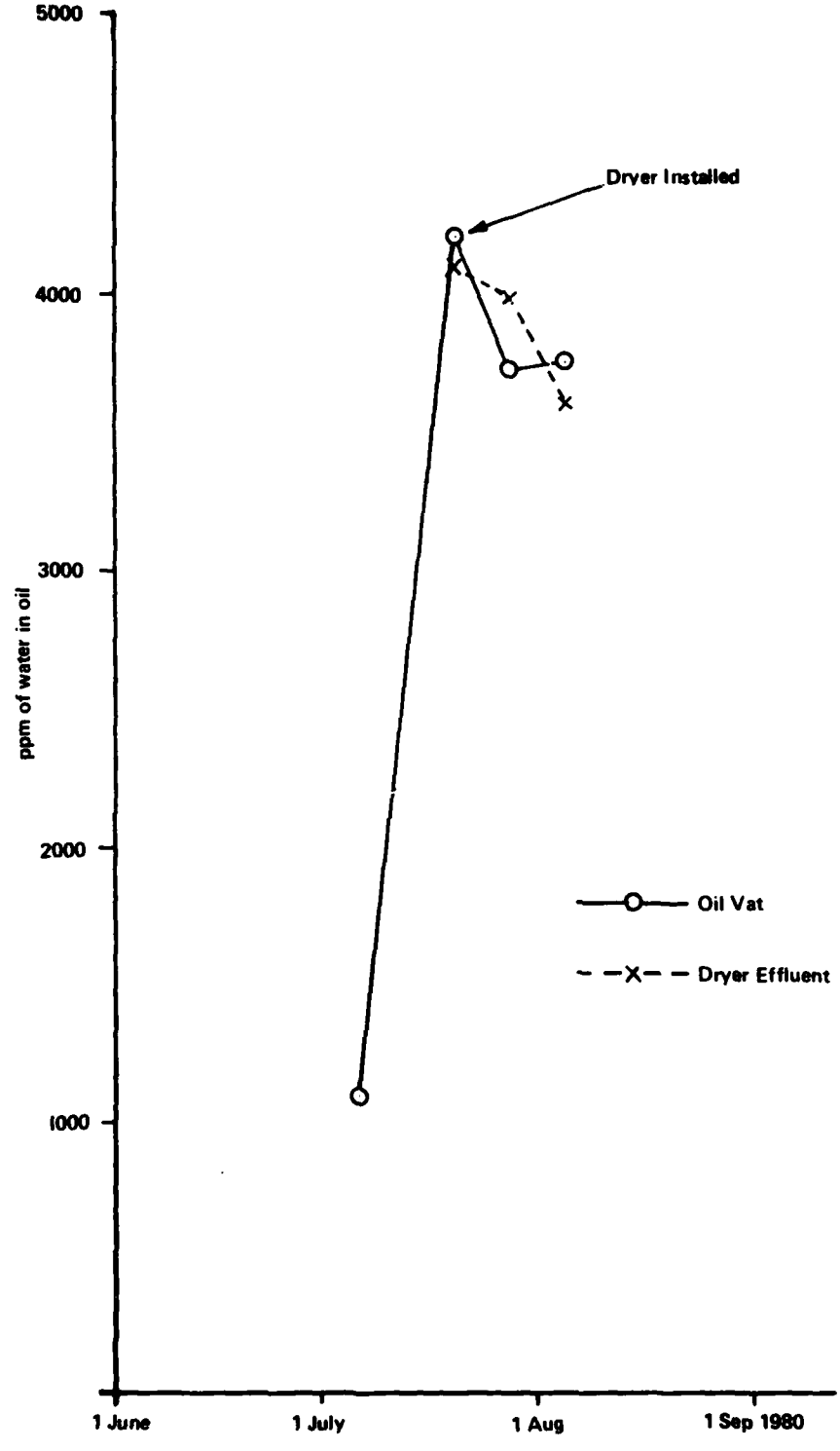

Figure 10. Water content of the castor oil.

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